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Thermal Effects on the Mechanical Properties of SiC Fiber Reinforced Reaction Bonded Silicon Nitride Matrix (SiC/RBSN) Composites

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SUMMARY

The elevated temperature four-point flexural strength and the room temperature tensile and flexural strength properties after thermal shock were measured for ceramic composites consisting of 30 vol % uniaxially aligned 142 μm diameter SiC fibers in a reaction bonded Si_3N_4 matrix. The elevated temperature strengths were measured after 15 min of exposure in air at temperatures to 1400 °C. The thermal shock treatment was accomplished by heating the composite in air for 15 min at temperatures to 1200 °C and then quenching in water at 25 °C. The results indicate no significant loss in strength properties either at temperature or after thermal shock when compared with the strength data for composites in the as-fabricated condition.

E-4375

INTRODUCTION

Recent studies[1,2] have shown that reinforcement of reaction bonded silicon nitride matrix (RBSN) by high strength, high modulus, chemically vapor deposited SiC fibers can yield a material which is stronger and tougher than unreinforced RBSN. These improvements in composite properties were obtained because of the use of SiC fibers that did not degrade or react with the matrix under thermal and environmental conditions associated with composite

fabrication. When stressed along the reinforcement direction at room temperature, this composite showed a stress-strain behavior mimicking to that of metals with the first matrix cracking corresponding to an apparent yield point.

A typical stress-strain behavior for the SiC/RBSN composite in tension, shown in Fig. 1(a), indicates three distinct regions. In the initial region OA, the stress increases linearly with strain. In the second region AB, the stress-strain curve became nonlinear due to the onset of matrix cracking normal to the loading direction. After a matrix crack appears, the applied stress momentarily decreases, resulting in a jog in the stress-strain curve. The frictional or mechanical bond formed between the fiber and the matrix allows the advancing matrix crack to deflect at the fiber/matrix interface without affecting the fiber properties. On the other hand, if the fiber and the matrix were strongly bonded, the composite matrix cracks would propagate directly through the fibers and the composite would fail catastrophically upon first matrix cracking, a behavior similar to that observed for the unreinforced monolithic ceramic. This latter behavior would show little improvement in toughness for the composite over a monolith.

With continued straining beyond point A, the matrix blocks on either side of the crack load up until they crack. Each matrix crack results in a jog in the stress-strain curve. The process of matrix cracking and load transfer between fiber and matrix continues until the matrix brakes into such small blocks that the fibers can no longer transmit the breaking stress to them. Beyond this stress level, the matrix does not carry any significant load and the matrix crack spacing reaches a limiting value.[1] In fact, from the measurements of the crack spacing, the matrix fracture stress, and the elastic properties of the components one can determine the average interfacial shear

strength between the fiber and the matrix. In the BC region of Fig. 1(a), the stress varies linearly with the strain until final fracture of the composite. Since the matrix contribution is negligible in this region, the deformation is controlled by the fiber properties.

When stressed in the flexural mode, the composite shows a deformation behavior similar to that observed in the tensile mode except for the absence of the second linear region, as shown in Fig 1(b). In the initial region, OA, the load-displacement behavior is linear. Beyond this region the stress-strain curve becomes nonlinear due to matrix macrocracking. At ultimate flexural stress the composite fails by successive fiber ply failure.

Because of their low density, high strength, and noncatastrophic failure behavior, the SiC/RBSN composites show significant potential for use in advanced high temperature structural applications. However, the major issue is whether the metal-like stress-strain behavior and excellent properties observed for the composites in the as-fabricated condition can be retained after exposure to environmental and thermal conditions similar to that of high performance engines or other applications. The effects of long term environmental exposure on the properties of the composites will be the subject of an another paper.[3] The objective of the present study was to determine the deformation and failure behavior for unidirectionally reinforced SiC/RBSN composites at temperatures to 1400 °C after 15 min exposure in air, and at room temperature after thermal shock quenching from temperatures to 1200 °C.

EXPERIMENTAL

Composite Fabrication

The block diagram shown in Fig. 2 describes the steps involved in the fabrication of SiC/RBSN composites. The starting materials used for composite fabrication were SiC fiber mats and silicon matrix cloth. The SiC fibers used

were double coated SCS-6 fibers obtained from Textron Specialty Materials Division. The composition and properties of this fiber are described elsewhere.[4] The SiC fiber mat was prepared by winding SiC fibers to the desired spacing on a cylindrical drum and coating them with a slurry of fugitive polymer binder and an organic solvent. After carefully drying the fiber mat, it was cut to the required dimensions. The silicon matrix cloth was prepared by using high purity silicon powder obtained from Union Carbide. The silicon powder was mixed with an additive (for enhancing nitridation), a fugitive polymer binder and an organic solvent until a dough was formed. The resultant dough was rolled to the desired thickness and cut to the desired dimensions.

The composite was fabricated by stacking alternate layers of SiC fiber mat and silicon cloth in a molybdenum die and hot pressing in vacuum or in a nitrogen environment at a suitable combination of temperature and pressure to produce a handleable preform. The details of the fabrication technique were reported elsewhere.[4]

The composite preforms were then transferred to a horizontal nitridation furnace consisting of a recrystallized Al_2O_3 reaction tube with stainless steel end caps. High purity nitrogen gas flowed through the furnace before, during and after nitridation. The nitridation of the composite preforms was accomplished between 1100 and 1400 °C, a temperature range similar to that employed for nitridation of the monolithic RBSN. The typical dimensions of a nitrided composite panel were 150 by 50 by 2.2 mm.

A photomicrograph of the polished cross-section of a typical 30 vol % SiC/RBSN composite specimen with an extra outer matrix layer is shown in Fig. 3. The composite matrix contained ~30 vol % porosity and displayed density variations around the fibers. The matrix density between layers of fiber plies

was significantly higher than between fibers within fiber layers. The average room temperature properties for this composite are shown in Table I.

Specimen Preparation

The as-fabricated composite panels were typically fabricated with an extra matrix layer on their top and bottom surfaces. This matrix layer was partially removed by grinding the composite panel on SiC abrasive papers. The specimens for mechanical property measurements were prepared from the composite panels by cutting and grinding with a diamond impregnated abrasive wheel. For elevated temperature four-point flexural strength measurements, typical specimen dimensions were 63.5 by 6.4 by 2.0 mm and the span to depth ratio (L/h) was 25. The four-point bend fixture was machined from hot pressed SiC material. The flexural test was conducted after 15 min of exposure in air.

For the thermal shock study, tensile specimens of dimensions 127 by 12.7 by 2.0 mm and flexural specimens of dimensions similar to those used for elevated temperature strength measurements were used. For thermal shock tests, composite specimens were heated in air for 15 min at temperatures between 300 and 1200 °C and then quenched in a water bath maintained at 25 °C. After drying for 8 hr in an oven set at 100 °C, the specimens were tested at room temperature. For tensile tests, specimens were adhesively bonded with glass fiber reinforced epoxy tabs at the specimen ends leaving 50 mm as the test gauge length. To monitor strains during tensile testing, wire wound strain gauges were attached to the central portion of the specimens.

All tensile and flexural tests were performed in an Instron machine at a crosshead speed of 1.26 mm/min. At each test condition the strengths of five specimens were measured.

RESULTS

Composite Mechanical Properties

Elevated temperature flexural strength. Typical load-displacement curves for the 30 vol % SiC/RBSN composites tested in the four-point flexural mode at 25, 1000, and 1400 °C are shown in Fig. 4. According to this figure, the three load-displacement behaviors were similar up to 100 Pa. Above this load level, the load-displacement curves became nonlinear. As the test temperature was increased the nonlinear region became more pronounced. The four-point ultimate flexural strengths for the composites as a function of temperature from 800 to 1400 °C are shown in Fig. 5. Each data point in Fig. 5 represents the average of at least five tests with the room temperature tests representing over 10 specimens. The error bars indicate one standard deviation. These strength values were determined from the ultimate load and elastic beam theory. However care must be exercised in interpreting the flexural strength data. For composites especially at high temperature and after matrix cracking these values probably underestimate the true tensile stress on the outer surface of the specimen. Nevertheless, these strength data can be used to measure the temperature dependence of the ultimate load carrying capability of the SiC/RBSN composites in bending.

According to Fig. 5, up to 1300 °C the ultimate flexural strength values were similar to those measured at room temperature. At 1400 °C however, a small loss in the ultimate flexural strength was observed possibly due to creep deformation of the matrix and the fibers. In fact above 1000 °C, the composite specimens did not fracture on stressing, but bent into a U shape and were still capable of substantial load bearing capability. Because of the large deflection associated with the specimens in this temperature range, the ultimate flexural strength values calculated based on elastic beam theory are

inaccurate. If stressed sufficiently to finally fracture, the specimens showed brushy, broomy fractures with a large amounts of fiber pullout. This is indicative of fairly weak fiber-matrix bonding.

In Fig. 6, the ultimate flexural strengths for the composite at room temperature, 1200, and 1400 °C are compared with similar data for hot pressed Si_3N_4,[5] monolithic RBSN,[5] and one-dimensional SEP SiC/SiC composite.[6] In comparing these data, we assumed these materials failed in the tensile mode. The data represented as bar graphs in the Fig. 6 indicate that SiC/RBSN composites show better strength at 1400 °C than any of the other compared materials in the fast fracture mode.

Thermal shock resistance. Thermal shock resistance for the uniaxially reinforced SiC/RBSN composites was determined by the water quench method. In this method, test specimens heated to predetermined temperatures were quenched in water maintained at 25 °C. The effect of quenching on the room temperature mechanical properties was evaluated. Figure 7 shows the matrix cracking stress and the ultimate strength data measured in the four-point flexural mode for the composites in the as-fabricated condition and after quenching from 300 to 1200 °C. Also shown in the figure for comparison purposes are the flexural strength data for monolithic RBSN quenched and tested under similar conditions. Figure 7 indicates that up to quenches from 400 °C, the flexural fracture strengths for monolithic RBSN are the same as the strength for the as-received untreated RBSN. Above this quench temperature, however, the monolithic RBSN strength decreased rapidly and then reached an approximately constant value of 35 MPa from 500 to 800 °C. In comparison, the composites quenched from 300 to 1200 °C showed no loss in the matrix flexural fracture strength from the as-fabricated condition, but the same composites showed an ultimate flexural strength loss behavior qualitatively similar to that observed for the quenched monolithic RBSN, but with the loss showing up at a 150 °C higher quench

temperature. Initially, the ultimate flexural fracture strength remained constant up to 600 °C. Above this temperature the ultimate flexural strength decreased to 450 MPa and then remained constant up to 1200 °C, the maximum quenching temperature used. On the other hand, according to Fig. 8, the quenched composite specimens tested in the tensile mode showed no significant loss in their matrix fracture strength, primary composite modulus, and ultimate strength values compared to that for the as-fabricated composites. From these results it is concluded that thermal shocking of composites had no significant influence on their room temperature axial tensile properties, but did decrease their ultimate flexural strength.

To determine the mechanism for ultimate flexural strength loss, the flexural specimens stressed to different stress levels between the matrix fracture stress and the ultimate fracture stress were unloaded and then examined under an optical microscope. The limiting matrix crack spacing, which is an indicator of interfacial shear strength and bonding between the fiber and the matrix, was measured and the failure mode of the composite was closely monitored. This examination revealed that the matrix crack spacing, and the matrix and ultimate failure behaviors for the specimens thermally shocked from temperatures between 300 and 600 °C were similar to that for the as-fabricated composites. On the other hand, the composites quenched from temperatures above 800 °C showed similar matrix crack spacing, but their failure behavior above matrix fracture was slightly different from that for the as-fabricated composites. These specimens showed failure starting from the tensile side with the onset of matrix cracks normal to the fibers. At higher stress levels, as shown in Fig. 9, irregular matrix cracks were formed. These irregular cracks did not exist at stress levels less than the matrix fracture stress. On the other hand, if these cracks were caused by thermally induced stresses during

the quenching operation, they would have lowered the matrix fracture stress and the primary modulus of the composites. The fact that quenched composites showed no loss in the matrix fracture strength or in the primary modulus proves that these cracks are formed during stressing at stress levels greater than the matrix fracture stress. With continued stressing, the failure behavior changed from tensile to shear delamination as shown in Fig. 10. Based on these observations and the tensile results we conclude that flexural strength loss in SiC/RBSN composites quenched from temperatures above 650 °C is probably caused by loss of interply integrity of the specimen after matrix fracture.

For monolithic ceramics, the critical quench temperature difference, ΔT, above which mechanical property degradation occurs after water quench tests can be estimated from the equation,[5]

$$\Delta T = \frac{\sigma(1 - \mu)}{\alpha E} \qquad (1)$$

Here σ is the ultimate matrix fracture stress, μ is Poisson's ratio, α is thermal expansion coefficient, and E is the Young's modulus. Using $E = 176$ GPa, $\mu = 0.22$, $\alpha = 2.29 \times 10^{-6}/°C$,[5] and $\sigma = 220$ MPa for monolithic RBSN from this study, the calculated critical temperature difference is 426 °C, whereas the measured value is 450 °C (cf Fig. 7). On the other hand substituting values of $E_c = 193$ GPa, $\mu_c = 0.21$, $\sigma = 227$ MPa, $\alpha_c = 4.0 \times 10^{-6}/°C$ for the SiC/RBSN composite in Eq. (1), the predicted critical temperature for strength degradation is 235 °C. Contrary to the prediction, the experimental data for the composites show no loss in matrix fracture stress. This suggests that the above equation based on the elastic properties and thermal expansion coefficient and derived for monolithic ceramics may not be applicable for composites. To understand their thermal shock behavior, other factors such as heat transfer characteristics and specimen geometry must be considered. In general the higher the thermal conductivity or the thinner the cross section of

the material is, the better is its thermal shock resistance. The fact that the thermal conductivity of SiC fibers, k_{SiC} = 0.050 cal/sec·cm^2·°C·cm is nearly twice that for the monolithic RBSN, k_{RBSN} = 0.030 cal/sec·cm^2·°C·cm, implies that the composite should have higher thermal conductivity and better thermal shock resistance than the unreinforced RBSN. However, to prove whether the superior thermal shock resistance seen for the composite is due to the fibers or is due to the specimen geometry needs further study.

SUMMARY OF RESULTS

The flexural strength at temperatures to 1400 °C after 15 min exposure in air and the room temperature strengths after thermal shock tests from temperatures to 1200 °C were measured for uniaxially reinforced SiC/RBSN composites. The important findings are as follows:

1. Under fast fracture, SiC/RBSN matrix composites retain their as-fabricated strengths to 1400 °C and show better elevated temperature strength than unreinforced RBSN, hot pressed Si_3N_4, or SEP SiC/SiC composites.

2. SiC/RBSN matrix composites show no loss in matrix fracture strength and ultimate tensile strength when quenched from temperatures to 1200 °C, but the composites show loss in ultimate flexural strength when quenched from temperatures above 600 °C, which is about 150 °C higher than unreinforced RBSN.

CONCLUSIONS

After short time exposures in air, the unidirectionally reinforced SiC/RBSN composites retained their high matrix fracture strength, pseudo-yield point, and noncatastrophic failure at temperatures to 1400 °C and at room temperature after thermal shocking from temperatures as high as 1200 °C. These superior properties coupled with their low density make SiC/RBSN a potential candidate for elevated temperature applications in advanced heat engine components or other high temperature structural applications.

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TABLE I. - ROOM TEMPERATURE PHYSICAL MECHANICAL PROPERTY DATA FOR ~30 VOL % SiC/RBSN COMPOSITES

Density, gm/cc	~2.3
Porosity, percent	~30
Elastic modulus, GPa	193±7
Four-point bend strength, MPa	
Matrix fracture stress	230+40
Ultimate strength	811+100
Tensile strength, MPa	
Matrix fracture stress	227±41
Ultimate strength	550+100

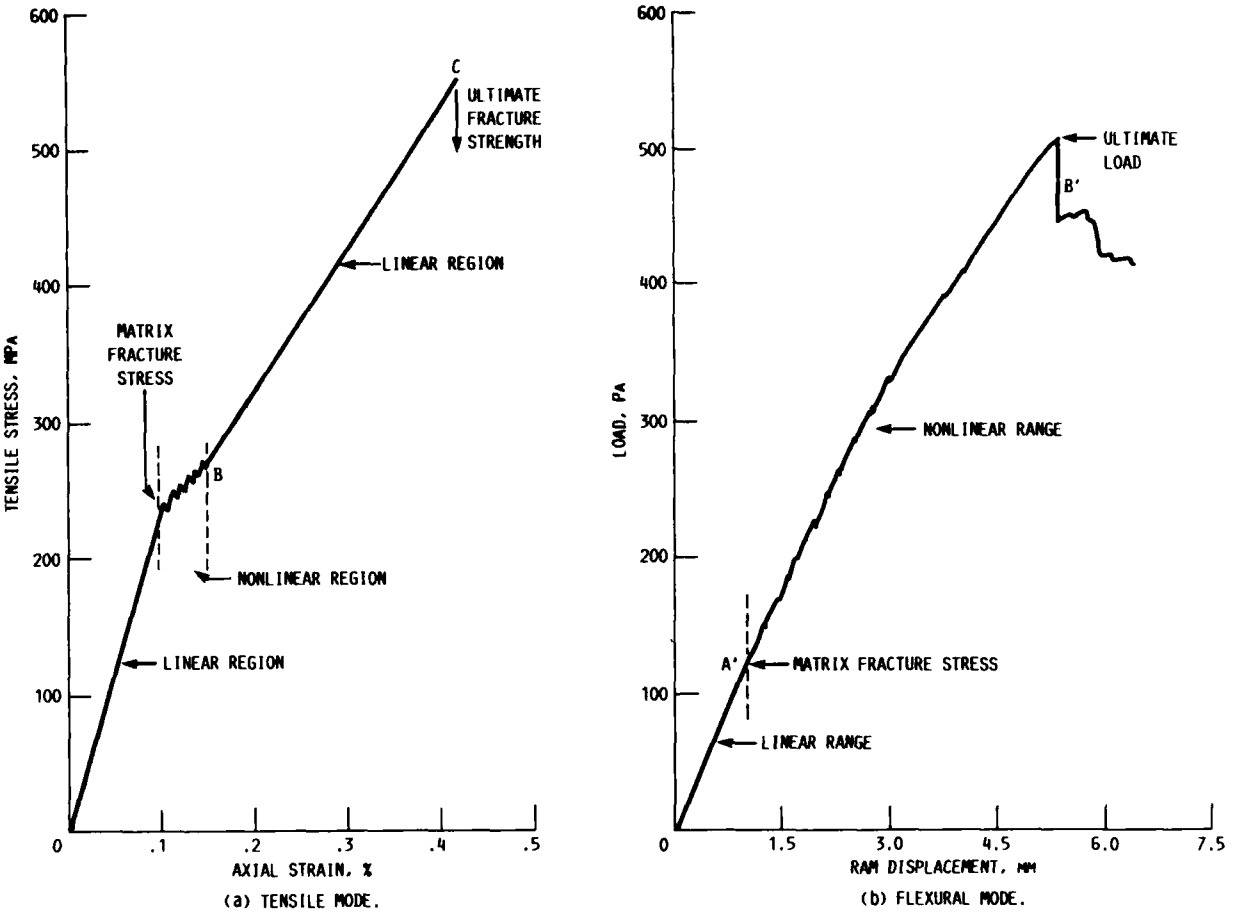

FIGURE 1. - TYPICAL DEFORMATION BEHAVIOR FOR *UNIDIRECTIONALLY* REINFORCED SiC/RBSN COMPOSITE.

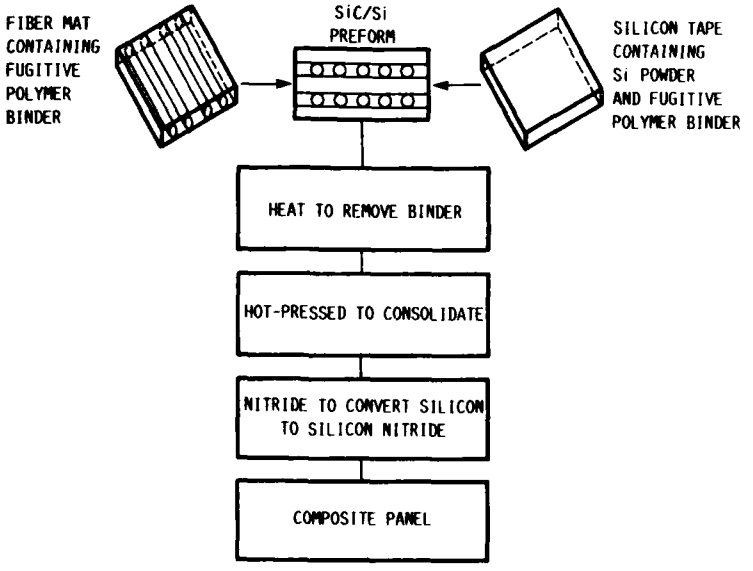

FIGURE 2. - BLOCK DIAGRAM SHOWING SiC/RBSN COMPOSITE FABRICATION.

FIGURE 3. - TYPICAL CROSS SECTION OF ~30 VOL % SiC/RBSN COMPOSITE.

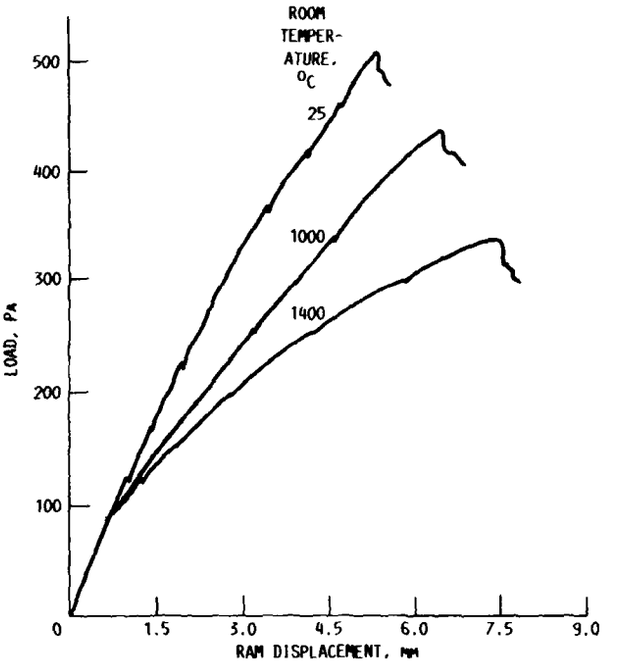

FIGURE 4. - TYPICAL LOAD-DISPLACEMENT BEHAVIORS IN 4-POINT BENDING FOR 30 VOL % SiC/RBSN COMPOSITES AT 25, 1000, AND 1400 °C.

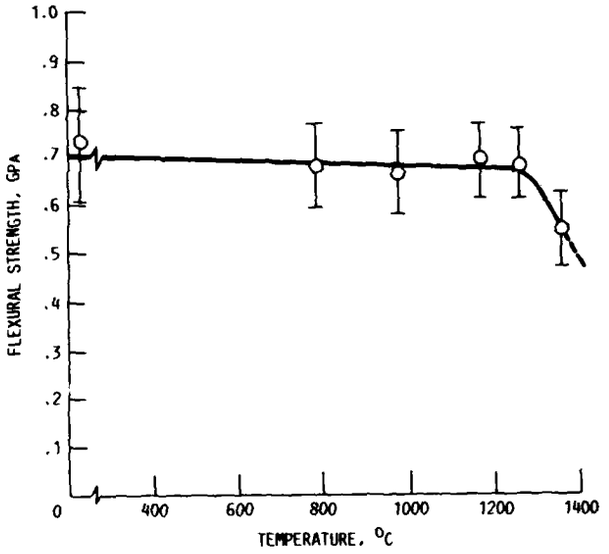

FIGURE 5. - ULTIMATE FLEXURAL STRENGTH AS FUNCTION OF TEMPERATURE FOR 30 VOL % SiC/RBSN COMPOSITES AFTER 15 MINUTES OF EXPOSURE IN AIR.

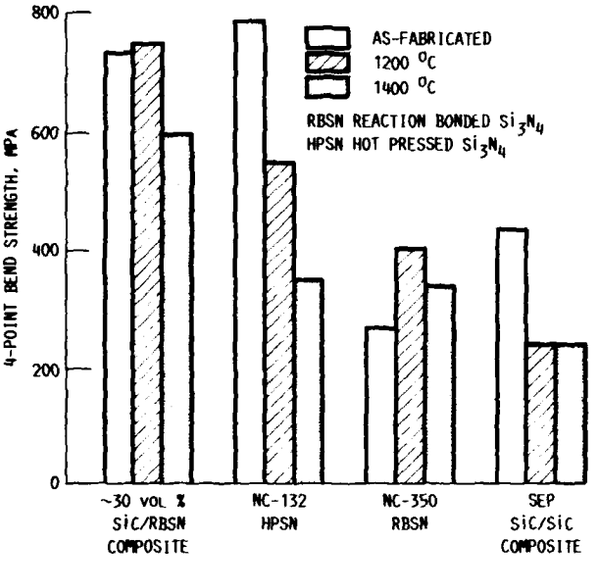

FIGURE 6. - BAR GRAPH SHOWING BEND STRENGTH FOR ~30 VOL % SiC/RBSN, HOT PRESSED Si_3N_4 (NC-132), RBSN (NC-350), AND 1-D SEP SiC/SiC COMPOSITE AT 25, 1200, AND 1400 °C.

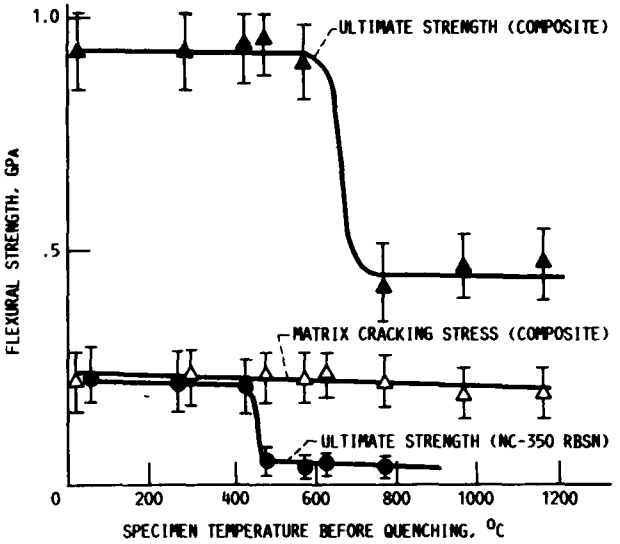

FIGURE 7. - ROOM TEMPERATURE 4-POINT FLEXURAL STRENGTHS FOR 30 VOL % SiC/RBSN COMPOSITES AND MONOLITHIC RBSN (NC-350) AFTER QUENCHING FROM INDICATED TEMPERATURES.

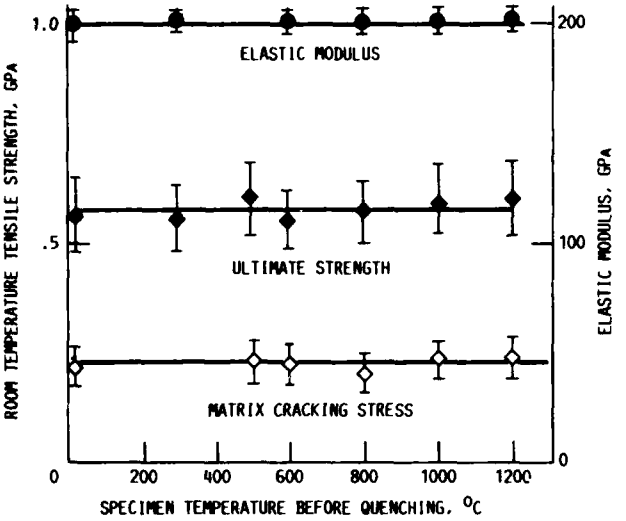

FIGURE 8. - ROOM TEMPERATURE TENSILE PROPERTIES FOR 30 VOL % SiC/RBSN COMPOSITES AFTER QUENCHING FROM INDICATED TEMPERATURES.

FIGURE 9. - A 30 VOL % SiC/RBSN COMPOSITE SPECIMEN QUENCHED FROM 800 °C AND THEN STRESSED IN 4-POINT BENDING ABOVE LINEAR REGION SHOWING IRREGULAR MATRIX CRACKING ON TENSILE SURFACE.

FIGURE 10. - A 30 VOL % SiC/RBSN COMPOSITE SPECIMEN QUENCHED FROM 800 °C AND THEN STRESSED TO 400 MPa IN 4-POINT BENDING SHOWING SHEAR DELAMINATION FAILURE.

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16. Abstract

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